THE APPLICABILITY OF STANDARD TEST METHODS TO THE ANALYSIS OF COAL SAMPLES FOR COAL RESEARCH

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INTRODUCTION

Standard test methods for routine coal analysis in the United States include those of the American Society for Testing and Materials (ASTM) and, with limited application, those of the International Organization of Standardization (ISO). The former consist of national standards used in the United States and Canada, while the latter have been developed by ISO member nations for international trade. While these methods are used throughout the coal industry and in commerce to establish coal quality, they may not be applicable in the analysis of coal samples for research purposes. One problem area is the chemical analysis of coal. This becomes particularly evident when using ASTM methods which were primarily designed for the analysis of bituminous coal to analyze lignetic and subbituminous coals. Here, researchers and analysts find themselves trying to fit a square peg in a round hole.

In this paper some of the difficulties that have been observed in our analysis of research coal samples for major, minor, and trace elements are emphasized, and suggestions for eliminating these problems are presented.

DISCUSSION

Moisture in the Analysis Sample

Determining the moisture in the analysis by weight loss at 104-110°C presents several problems, especially if the current ASTM method D3173 (1) is used. In our experience, the ASTM method is unsuitable for low-rank coals because it does not use an inert gas as the purge gas and recommends too short a drying period. Oxidation -- gain in weight as the sample reacts with oxygen in the air -- can take place, and not all of the 104-110°C moisture is removed during the recommended one-hour drying time. In addition, with certain low-rank coals, decarboxylation during the drying period can result in weight loss. At present, our use of moisture data is primarily for calculation of sulfur, ash, and trace element data to moisture-free bases, so a modified ASTM method similar to the ISO method 331 (2) is used. The coal sample is heated in an oven at 104-110°C for three hours. The oven is purged with dry, purified, and preheated nitrogen.

Ash in the Analysis Sample

The ash in coal is the noncombustible residue that remains when coal is burned. In the ASTM method D3174 (3), the coal sample is placed in a cold furnace and heated gradually so the temperature reaches 450 to 500°C in one hour and 700 to 750°C at the end of the second hour. The ISO method 1171 (4) recommends a 815°C final temperature. In both methods the sample is ignited at the appropriate final temperature to constant weight.

For our research samples, we have selected a procedure in which the coal samples are placed in a cold muffle furnace; the temperature is incremented at the rate of 100°C per hour until the final temperature is attained. Although slower, this procedure prevents ignition of the coal, which can result in the physical loss of material. In addition, this slower rate minimizes sulfur retention in the ash.

Ash Analysis - Major and Minor Ash Elements

The analysis of coal ash for major and minor elements is important for determining the ash chemistry. The current ASTM method D3682 (5) requires fusion of the sample with lithium tetraborate followed by analysis by atomic absorption spectrophotometry (AAS). Presently the list of routinely determined ash elements includes Si, Al, Fe, Ti, Ca, Mg, Na, K, P, and S. For our research samples a more complete ash characterization is necessary so that elements such as Sr, Ba, Mn, and Zn are also determined. In addition, methods utilizing mixed-flux fusions are being evaluated to eliminate the need to use one flux (lithium metaborate) for highly siliceous ashes and another flux (lithium tetraborate) for ashes containing high contents of iron oxides. These aspects are being addressed because of the extreme variability in the ash chemistry encountered when analyzing lignite ash versus bituminous coal ash.

Although AAS is the technique utilized in the ASTM standard method, the determination of additional elements, coupled with the need to reduce analysis turnaround time, has prompted the evaluation of multielement sequential and/or simultaneous determination systems such as inductively coupled plasma-atomic emission spectrometry (ICP-AES) for coal ash analysis.

Ash Analysis - Trace Elements in Coal Ash

The determination of trace elements in coal ash is relatively straightforward and will be indicative of the trace element content of the coal if the particular trace element is not volatilized during ashing. The current ASTM method D3683 (6) utilizes a 500°C ash for the determinations of Be, Cu, Cr, Mn, Ni, Pb, V, and Zn by flame atomic absorption spectrophotometry. While flame AAS is satisfactory for most of the trace elements mentioned, many coal ashes contain levels of Pb and V that are just above the flame AAS detection limits; hence, quantification for those elements is difficult. Flameless AAS is being evaluated as an alternative for the Pb determination and ICP-AES is being evaluated for the V determination as well as for the other trace elements listed above.

Analysis of Coal for Major, Minor, and Trace Elements

As mentioned previously, if the inorganic components present in the coal are not volatilized during ashing of the coal, and the ash content is known, these elements can be determined in the ash and calculated to a coal basis. Unfortunately, the literature reports situations where selected elements in coal, such as sodium, lead, and cadmium, are volatilized during ashing (7,8,9). Situations such as these cast doubt on the universal applicability of elemental analysis methods for coal that require muffle furnace ashing.

Wet ashing techniques include the use of mixtures of perchloric and other acids for the dissolution of coal. While wet ashing minimizes the risk of volatilization of the major, minor, and trace elements, most coal analysis laboratories avoid the use of perchloric acid. Other approaches of coal sample preparation for analysis such as microwave oven digestion and slurry techniques exhibit contamination problems and/or low recoveries for many elements.

We recommend the use of nitric-perchloric acid digestions (10) and oxygen bomb combustions (11) in the preparation of coal samples for spectrochemical analysis. These procedures eliminate any ambiguity associated with potential elemental losses by volatilization during the sample preparation.

CONCLUSIONS

In analyzing coal research samples for their chemical composition, it is apparent that certain current standard test methods require modification or they are not applicable. Coal research, and possibly most new uses of coal, will require higher standards of quality control and accuracy than are currently quoted in many of the existing standard test methods. These factors will become increasingly more important when economic decisions must be made based on the validity, i.e., accuracy, of coal analysis data.

ACKNOWLEDGMENT

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QUANTITATIVE ASPECTS IN CP/MAS EXPERIMENTS ON WHOLE COALS AND MACERALS*

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Introduction

Solid-state NMR spectra are now being obtained routinely for a wide variety of fossil-fuel materials, including coals, using cross-polarization (CP) techniques combined with high-power decoupling and magic-angle sample rotation (MAS).[1] However, the quantitative reliability of CP/MAS experiments on coals has recently received considerable attention.[2-8] Because coals are heterogeneous by nature, a single CP experiment can give inadequate quantitative information and may be very misleading. Problems can arise because the efficiency of cross polarization to different carbons in the sample depends upon their characteristic polarization transfer times (T_{CH}) and on the behavior of their respective proton reservoirs with regard to spin-lattice relaxation in the rotating frame ($T_{1\rho}^{H}$). With regard to the analysis of coals, there is the additional complication that carbons in the vicinity of free radicals may not be detected due to dipolar interactions.

The purpose of the present investigation is to identify the important experimental parameters which govern NMR signal intensities in solid-state experiments on whole coals and separated coal macerals and, subsequently, to devise computer-assisted methods which allow absolute signal intensities to be calculated from the data. New methods have also been developed to evaluate the number of carbon spins that are detected in solid-state NMR experiments. Any missing carbon signal intensity has been attributed to the presence of paramagnetic centers or to inefficient carbon polarization.

Experimental

Solid-state ^{13}C spectra were obtained at 25.18 MHz on a Bruker CXP-100 spectrometer with a doubly-tuned single coil probe and a dual air-bearing spinning apparatus. The spinners were made of ceramic with an internal volume of 0.3 ml and were spun at approximately 4 kHz. Relaxation time experiments were carried out employing contact times between 0.05 and 10 ms, a 2 s pulse repetition rate, and a 67 kHz proton decoupling field. Carbon signal intensities were determined for aromatic (110-160 ppm) and aliphatic (0-60 ppm) absorption bands. For the aromatic carbons, signal intensities of the spinning sidebands were added to the intensity of the centerband. The fit of the contact-time magnetization curves was obtained using a non-linear least squares computer program developed in these laboratories. Typically, 11-22 contact times were selected for a single analysis.

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Carbon spin–counting experiments were carried out by physically mixing approximately 12 percent hexamethylbenzene (HMB) by weight with the sample to be measured. Experimental conditions used for the measurements were a contact time of 4 ms, a 3 s pulse repetition rate, a 50 ms acquisition time, and a 67 kHz proton decoupling field.

The five maceral samples studied were a resinite from Utah, Blind Bear Canyon mine, a sporinite isolated from PSOC 828, two vitrinites isolated from Illinois No. 2 hvC bituminous and PSOC 1103 coals, and a fusinite isolated from Illinois No. 2 hvC bituminous coal. The coal samples studied were a Victorian Brown coal (pale lithotype) from Australia, a Wyoming lignite, an Illinois hvC bituminous coal (Herrin No. 6), and a medium–volatile bituminous coal (PSOC 403). Analytical data for the separated coal macerals have been presented elsewhere.[9] The samples designated PSOC were obtained from the Penn State Coal Sample Bank.

Results and Discussion

Conventional CP/MAS experiments with a contact time of 1 ms initially were performed on the Illinois No. 2 vitrinite sample in order to establish which instrumental parameters were critical for quantitative analysis. Varying the proton decoupling field from 40–80 kHz or changing the pulse repetition rate from 0.5–3 s had little effect on the derived carbon aromaticity values. However, even a slight misadjustment of the Hartmann–Hahn matching condition resulted in a substantial reduction in the observed carbon aromaticity.

In a second set of experiments, the variation of the aromatic and aliphatic carbon signal intensities with contact time was investigated. Previous studies on coals have shown that treating all aromatic or all aliphatic carbons as having single relaxation behavior provides a reasonable model for computational analysis of the data.[4] Figure 1 shows the magnetization curves for four maceral samples. Absolute values for the carbon intensities (M_0) then are calculated by fitting the variation in carbon magnetization to equation 1 below:

$$M = M_0 \exp(-t/T_{CH}) \; (\; 1 \; - \; \exp(-bt/T_{1\rho}{}^{H}) \;), \qquad (1)$$

$$\text{where } b = 1 \; - \; T_{CH}/T_{1\rho}{}^{H}.$$

Our studies on model polymers have demonstrated that computed intensities for different carbon functional groups from variable contact–time experiments are accurate to within 3%. Calculated carbon aromaticities in Table 1 for a series of whole coals and maceral concentrates show the expected trends: aromaticity increases with increasing rank of the coals and, for macerals, in the order resinite < sporinite < vitrinite < fusinite. More importantly, each maceral sample in Figure 1 exhibits a unique, intensity response profile to the variation in contact time. The plots for the various macerals indicate that the intensity maxima of the aromatic carbons occur typically at longer contact times than those of aliphatic carbons. Hence, to select a single contact time which gives representative aromatic–aliphatic intensity ratios for the entire suite of macerals is difficult, if not impossible. Consequently, differences in relaxation behavior from sample to sample

are hard to reconcile from a single cross–polarization experiment and can lead to significant errors in the estimation of aromaticity values. The advantage of the computational method lies in its ability to determine carbon intensities which are independent of relaxation effects.

Carbon–spin counting experiments on the coal and maceral samples involve physically mixing a suitable intensity reference, hexamethylbenzene (HMB), with the sample to be measured. The ratio of the total carbon magnetizations of the sample over the reference calculated using equation 1 and normalized with respect to weight–percent carbon gives a good estimate of the proportion of carbon spins detected for the sample. Comparing results from Bloch–decay (SPE) and CP experiments allows one to distinguish what portion of the undetected signal intensity is due either to the presence of paramagnetic species in the sample or to inefficient carbon polarization. Figure 2 shows the results from a CP experiment on a resinite and a vitrinite sample, each containing approximately the same weight percent of HMB. The sharp signals at 20 and 135 ppm in each spectrum respectively represent the methyl and aromatic carbons of HMB. Visual inspection of the overall signal intensities of the two samples (in relation to the reference) reveals that a substantially lower number of carbons are detected for the vitrinite sample. When the integrated intensities are mathematically corrected for relaxation effects and compared to those calculated for the standard, the results indicate that 70% of the carbons in the resinite sample are being detected and only 35% for the vitrinite. These can be compared with values of 70% and 50%, respectively, obtained from Bloch–decay experiments. Therefore, one half of the carbons in the vitrinite sample are not observed due to paramagnetic line–broadening effects, while another 15% go undetected due to inefficient cross polarization. Moreover, the vitrinite data suggest that cross–polarization experiments largely discriminate against aromatic carbons, and thus, they provide minimum values for carbon aromaticity. The general trend observed for the entire suite of coal and maceral samples presented in Table 2 is a decrease in detected carbons with increasing carbon content of the sample.

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Table 1. Carbon Aromaticities(f_a) Derived from Contact Time Experiments using Equation 1.

Sample	f_a
Resinite(Hiawatha)	0.16
Sporinite(PSOC 828)	0.56
Vitrinite(Ill No. 2)	0.70
Vitrinite(PSOC 1103)	0.69
Fusinite(Ill No. 2)	0.82
Australian(Pale Lith)	0.37
Wyoming Lignite	0.55
Herrin No. 6(HVC Bit)	0.67

Table 2. Carbon Spin–Counting Experiments

Sample	% C Observed SPE[a]	% C Observed CP[b]	% C[c]
Resinite(Hiawatha)	70	70	83.8
Vitrinite(Ill No. 2)	50	35	72.0
Fusinite(Ill No. 2)	43	26	79.3
Wyoming Lignite		56	66.4
Herrin No. 6(HVC Bit)		55	62.8
MV Bit(PSOC 403)		40	78.0

[a] SPE = single–pulse excitation.

[b] CP = cross polarization.

[c] Carbon % on dmmf basis.

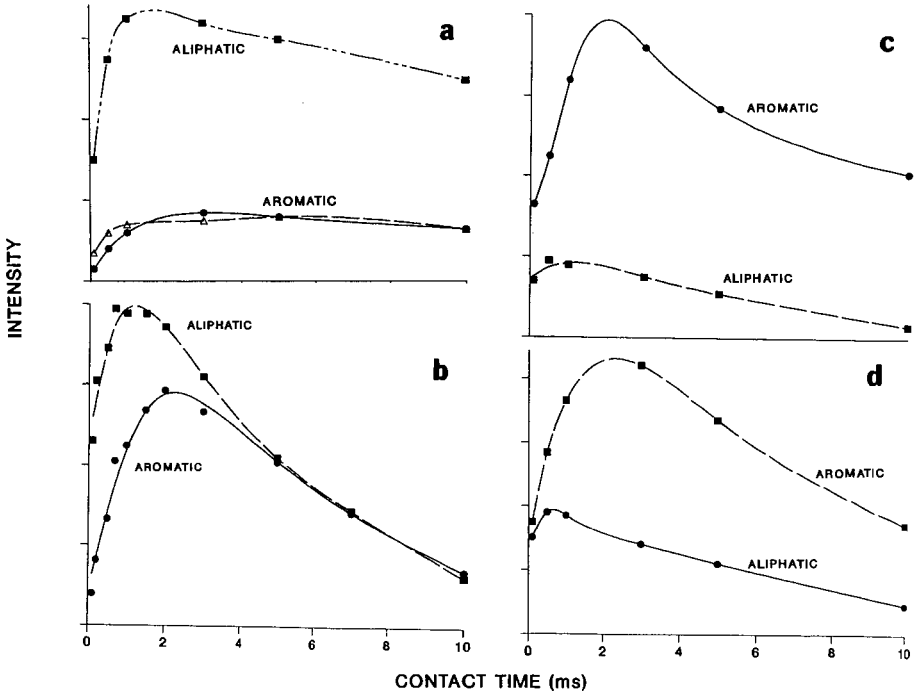

Figure 1. Variation in Carbon Signal Intensity with Contact Time for Maceral Concentrates a)Resinite, b)Sporinite, c)Fusinite and d)Vitrinite.

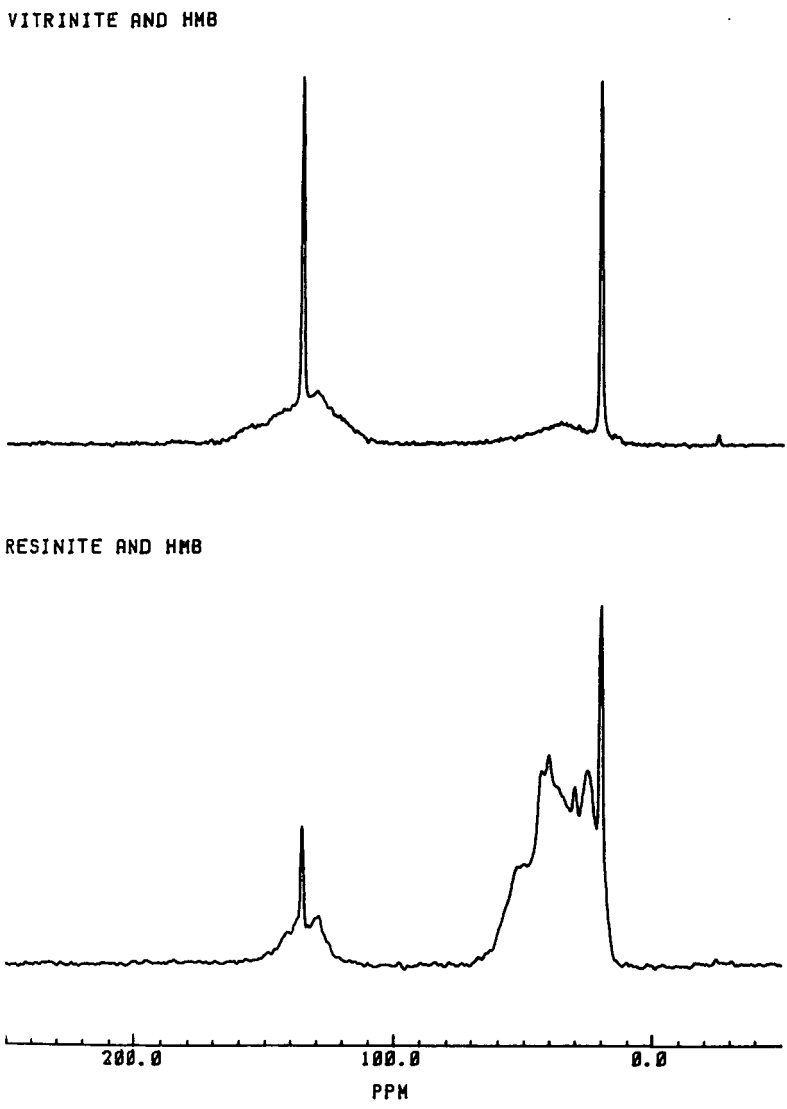

Figure 2. CP/MAS ^{13}C–NMR Spectra of Vitrinite and Resinite Samples with added HMB as the Intensity Standard.

STATUS OF THE PREMIUM COAL SAMPLE PROGRAM AT THE ARGONNE NATIONAL LABORATORY

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PURPOSE OF THE PREMIUM COAL SAMPLE PROGRAM

The purpose of the Premium Coal Sample Program is to provide the coal science research community with long term supplies of a small number of premium coal samples that can be used as standards for comparison and correlation. The premium coal samples produced from each coal and distributed through this program are chemically and physically as identical as possible, have well characterized chemical and physical properties, and will remain in a pristine condition over long periods of time.

The need for a Premium Coal Sample Program was expressed at the Coal Sample Bank Workshop held March 27 and 28, 1981 in Atlanta, Georgia.

WHAT A PREMIUM SAMPLE IS

A premium coal sample has been specially selected, processed and stored to keep it as close to its original condition as possible. Specifically: contact with oxygen has been minimized at all stages from mining, transport and processing in a nitrogen filled facility to sealing in amber colored glass vials. Relative humidity and temperature are controlled in the processing facility to maintain the equilibrium moisture of the original coal. Uniformity of samples is achieved by processing about 750 kg of coal in a single batch, mixing thoroughly in a special blender, and finishing with a spinning riffler to assure well-mixed samples. Activation analyses have confirmed the thoroughness of the mixing. Stability of the samples is maximized by sealing in amber-colored glass with a fuel-rich hydrogen-oxygen flame. Secure, long-term supplies result from an initial production of 10,000 five gram ampoules and 5,000 ten gram ampoules with 50 five gallon sealed glass carboys in reserve for future ampoule production from each metric ton sample of coal. Some special needs can be met from lumps stored in argon in two reserve 55 gallon drums, and two 15 gallon drums as part of the original sample. A separate nitrogen filled glove box will be used for processing these requests.

SELECTION, SAMPLE COLLECTION, AND TRANSPORT

Initially the coals have been selected to cover a wide range of chemical composition. The samples will include low-, medium- and high-volatile bituminous coals as well as lignite and sub-bituminous. These samples are channel-type samples, representing a uniform cross section of the seam from top to bottom. Sample collection, under the supervision of coal geologists from the U. S. Geological Survey, involves removal of coal up to 6" lumps from a freshly exposed face to special double plastic bags, transfer to stainless steel drums in a refrigerated semi-trailer at the surface, purging of samples in the drums with argon, and immediate transport to the processing facility at Argonne National Laboratory (ANL). A careful description of the geology of

the sample area and location will be prepared and available as a referenceable document.

SAMPLE PROCESSING

The processing facility is a large glove box made of aluminum panels about 12' tall, 4-5' wide and about 40' long. Clear plastic windows containing 70 pairs of rubber gloves permit observation and manipulation of the sample or equipment. During processing the box is filled with nitrogen and the oxygen concentration is kept at or below 100 ppm. The humidity is kept as high as possible but low enough to avoid condensation on the windows during operation. At the processing facility, the stainless steel drums are weighed and loaded into an airlock, which is then purged with nitrogen. The drums are emptied by means of a hydraulic drum dumper into a crusher which reduces the particle size to -1/2". The sample is then pulverized in an impact mill to obtain -20 mesh material. The pulverized material is collected in a nitrogen filled mixer-blender selected for gentle but thorough mixing. After thorough mixing the pulverized coal is conveyed to a spinning riffler and sealed in 10 gram ampoules and 5 gallon glass carboys or special 5 gallon transfer containers. The contents of the transfer containers are then recycled to the pulverizer and crushed to pass a 100 mesh screen. After thorough blending this material is conveyed to the packaging unit for sealing in 5 gram amber colored ampoules and 5 gallon borosilicate glass carboys. Figure 1 is a block diagram of the coal sample preparation.

CHARACTERIZATION

The coals are analyzed for three purposes: (1) homogeneity testing, (2) characterization, and (3) stability monitoring. Results are available for each coal in the form of a printed sample announcement. Requests to be placed on a mailing list should be sent to the author. The requestor should include mailing address, telephone number, and research interests.

Homogeneity testing includes sampling the product flow into carboys and ampoules throughout the processing to obtain 39 representative samples. These are placed in polyethylene containers for irradiation at the the University of Illinois TRIGA reactor. The samples are counted at ANL to monitor the Na, K and As activities. Other techniques are being evaluated to complement these measurements. After the results are analyzed and found to be satisfactory an announcement of the availability of samples is sent to individuals on the mailing list.

Characterization includes the efforts of over 70 different laboratories to establish physical and chemical properties of the samples. This number is desirable to permit a statistical analysis of the results. The analyses will include proximate, ultimate, calorific values, sulfur forms, equilibrium moisture, maceral analysis, Gieseler plasticity for the bituminous coals, and mineral matter major elements among others. Round robin analyses have been organized.

A variety of stability monitoring tests are used including evolved gas analysis and slurry pH. The gas analysis includes determinations of oxygen and light hydrocarbons to follow any possible diffusion of gas into the ampoules and diffusion of volatiles from the sample. The slurry pH monitors

the change in pH of the filtrate from a slurry of sample with distilled, deaerated water to monitor oxidation and release of products of pyrite decomposition. Sulfate ion concentration is also determined. In addition the bituminous samples will be monitored by repetitive Gieseler plasticity analyses.

AVAILABILITY AND DISTRIBUTION

Samples are made available to research personnel at a nominal replacement cost. A special glove box filled with nitrogen is available to transfer contents of ampoules to special sample holders on request. Also, a very limited quantity of lump coal, stored under similar inert conditions will be available on special request for special physical property measurements. The processing facility can be made available for occasional processing of special samples.

Orders are placed on the forms sent with the announcement of availability of the samples with the author. Prepayment is requested. Samples are available in packages of 6 ampoules of one size. Special cartons are used to assure safe delivery. Samples are shipped by United Parcel Service (UPS).

FIRST SAMPLE

The first sample, a medium volatile bituminous coal, was collected from the Lucerne #6 mine, owned by the Rochester and Pittsburgh Mining Company of Indiana, Pennsylvania. The sample was collected from the Upper Freeport seam near Homer City, Pennsylvania. A wedge shaped block was exposed by a continuous miner and used for the channel type sample.

INFORMATION ON SAMPLES

Each recipient of samples is asked to provide either a literature reference to papers in widely circulated journals, or a copy of less widely circulated public reports and papers, to be shared with other users of the samples. Listings of these references will be available on request to the author (phone 312-972-7374) either in printed versions or via computer terminal. The Premium Coal Sample Program expects to work with other coal sample programs in providing samples and sharing information.

Following the reports from the use of of a number of samples, workshops are planned to facilitate sharing research results and to foster basic understanding of the chemistry and physical properties of the coal. The first is expected to be scheduled in 1987 at ANL.

USERS ADVISORY COMMITTEE

A Users Advisory Committee provides useful suggehtions to the Program Manager. This group includes Drs.: Blaine Cecil, U. S. Geological Survey; Marvin Poutsma, Oak Ridge National Laboratory; Ronald Pugmire, University of Utah; William Spackman, Pennsylvania State University; Irving Wender, University of Pittsburgh; Randall Winans, Argonne National Laboratory; John Young, Argonne National Laboratory.

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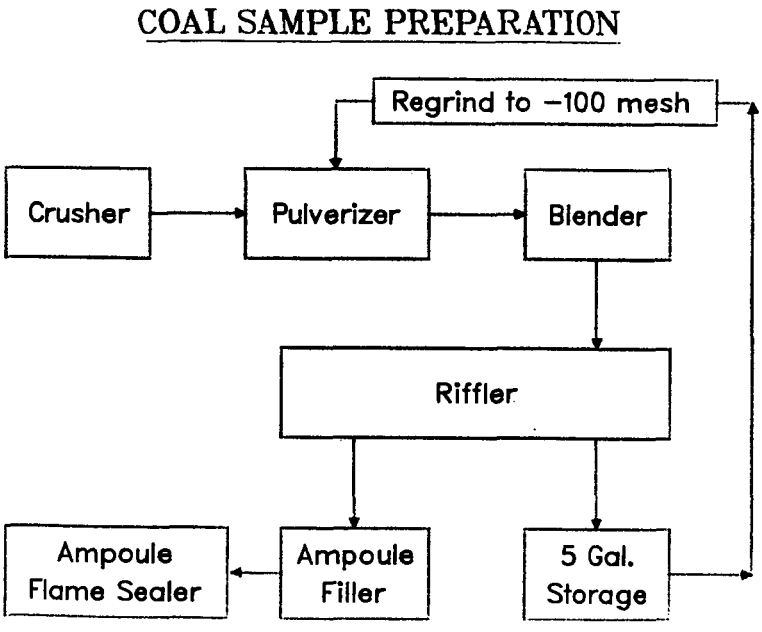
FIGURE 1



IDENTIFICATION OF OIL SPILLS BY FIELD IONIZATION MASS SPECTROMETRY

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The U.S. Coast Guard provided SRI with a sample of an oil spill and seven other samples from the bilge of various ships suspected to be the source of the spill. Field Ionization Mass Spectrometric (FIMS) analysis of the samples links the unknown X with Sample B for following reasons.

Field ionization of most organic substances results in the formation of their molecular ions only, i.e., no fragmentation except in a few cases. The mass spectrum is therefore a molecular-weight profile of the sample. In the attached spectra, we can see that all samples contain a bimodal distribution of molecular weights. In all samples, there is some material in the mass range 120 to 350 amu showing a maximum around 170 amu, most likely the fuel. There is also some material of higher molecular-weight material and a broad mass distribution ranging from about 300 to 800 amu, probably some lubricant or wax.

All organic compounds containing carbon, hydrogen, and oxygen have even molecular weights, and therefore a FIMS of a fuel will have a preponderance of intensities of even over odd masses. Peaks at odd m/z arise due to (i) natural ^{13}C abundance, (ii) molecules containing odd number of nitrogen atoms, and (iii) fragmentation. In field ionization there is very little fragmentation, and the attached spectra have all been corrected for natural ^{13}C abundance. For these reasons, the spectra show peaks mainly at even masses, and the few odd mass peaks generally correspond to nitrogen-containing compounds.

FIMS of the unknown sample was obtained in duplicate to indicate the reproducibility of the technique for these samples. As can be seen, the technique displays remarkable reproducibility. For the purpose of identifying the oil spill, we make use of three spectral features.

1. Relative intensities of the lighter and heavier material, fuel, and lube. Of course, weathering will alter the ratio, and this criterion must be used with some care. It is, however, safe to assume that any weathering will only increase the relative amount of the heavier material and not vice-versa.

   Applying this criterion, we can rule out samples F and G as being the source of the spill.

2. Pattern of intensities of some of the prominent peaks over a narrow mass range such that any weathering would alter the peaks to similar extents. Thus, examining the intensities of the peaks in the range 156 to 184 amu, paying particular attention to the pairs of peaks at m/z 156/160, 170/174, and 182/184, we can rule out samples A, D, E, and F. Samples F and G show a significantly more intense peak at m/z 146 relative to the one at m/z 142 than in other samples. This leaves samples B and C as possibly being linked to the spill.

3. Pattern of intensities of the odd mass peaks. These are the minor nitrogen-containing components that provide an additional fingerprint of the fuel. On the spectra, these peaks can be recognized as the dark stubs near the base line.

   On the basis of this criterion samples A, C, E, F, and G can be ruled out. The FI mass spectra of all the samples have also been plotted over the mass range 100 to 300 at much reduced full-scale intensity to better show the minor peaks and allow an easy comparison of the pattern of the odd mass peaks. Following this exercise, one arrives at the same conclusion.

The only sample not ruled out thus far is B.

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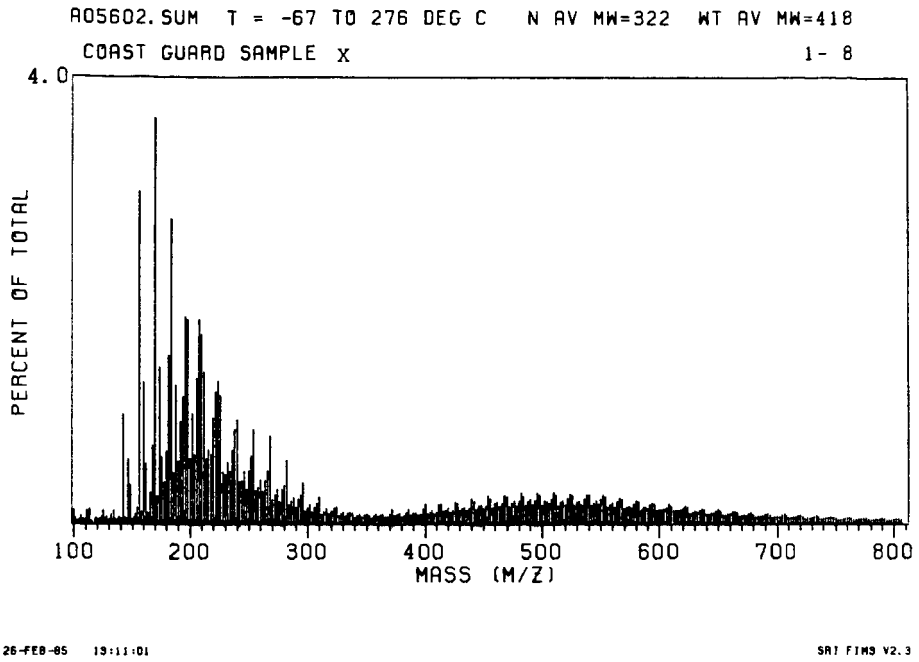
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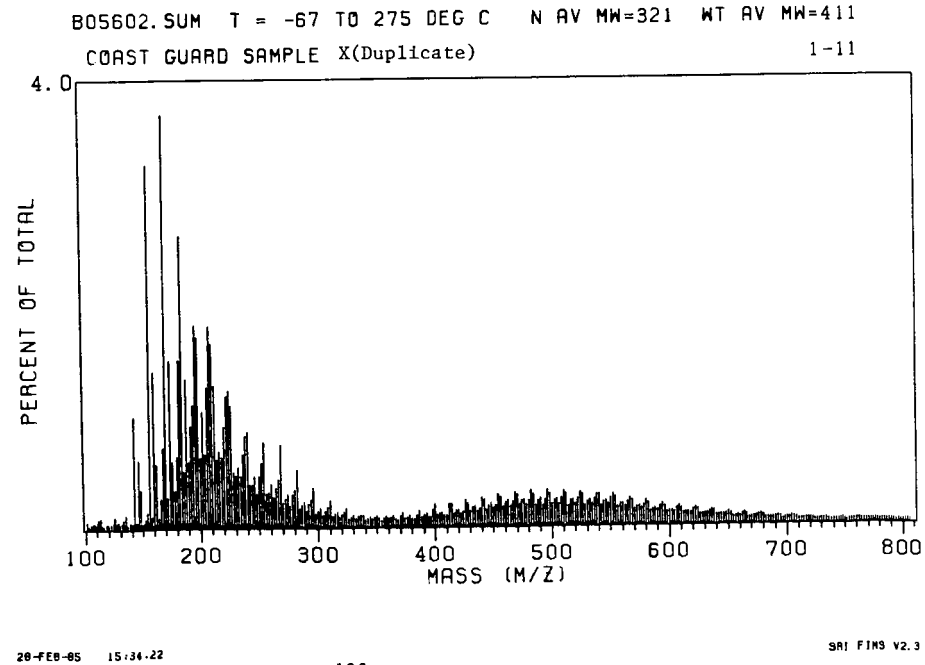
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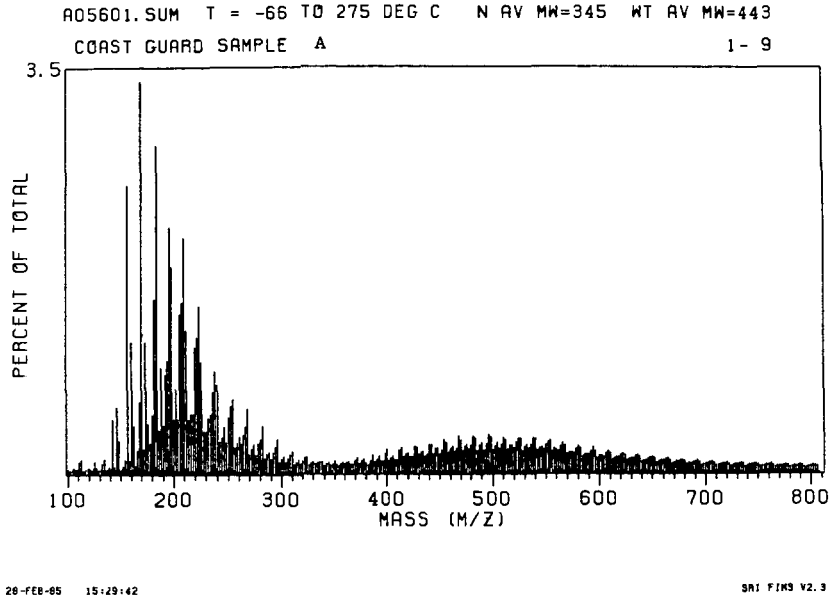
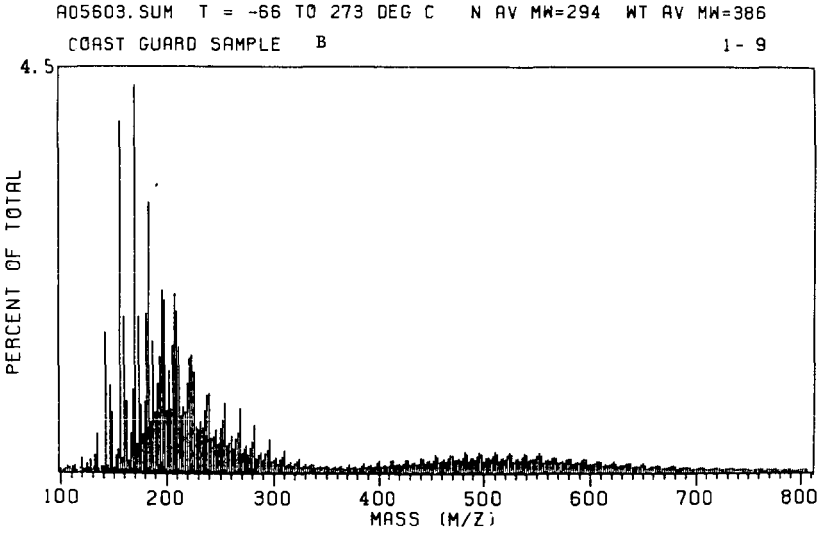
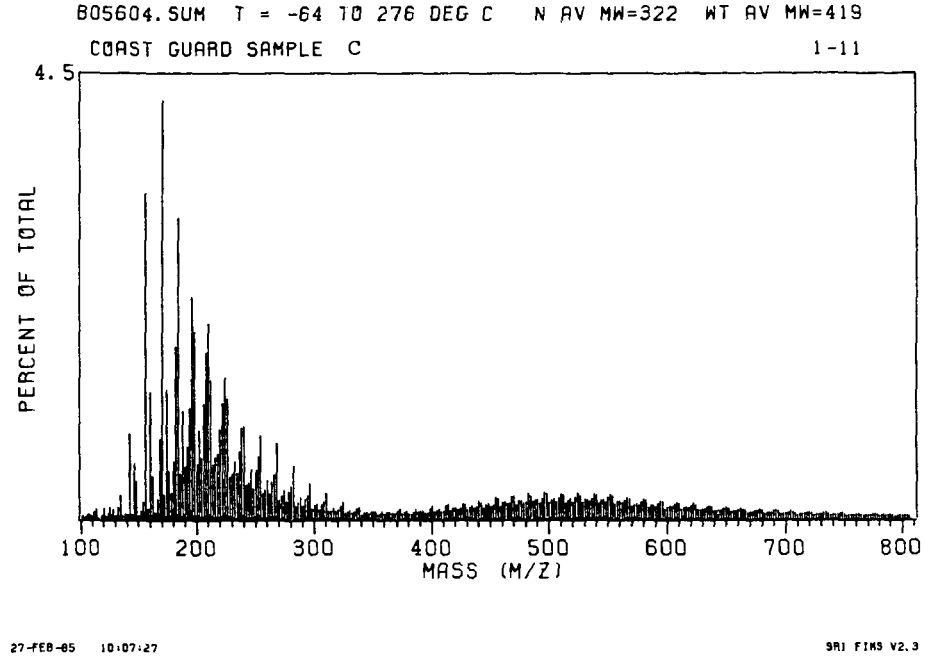
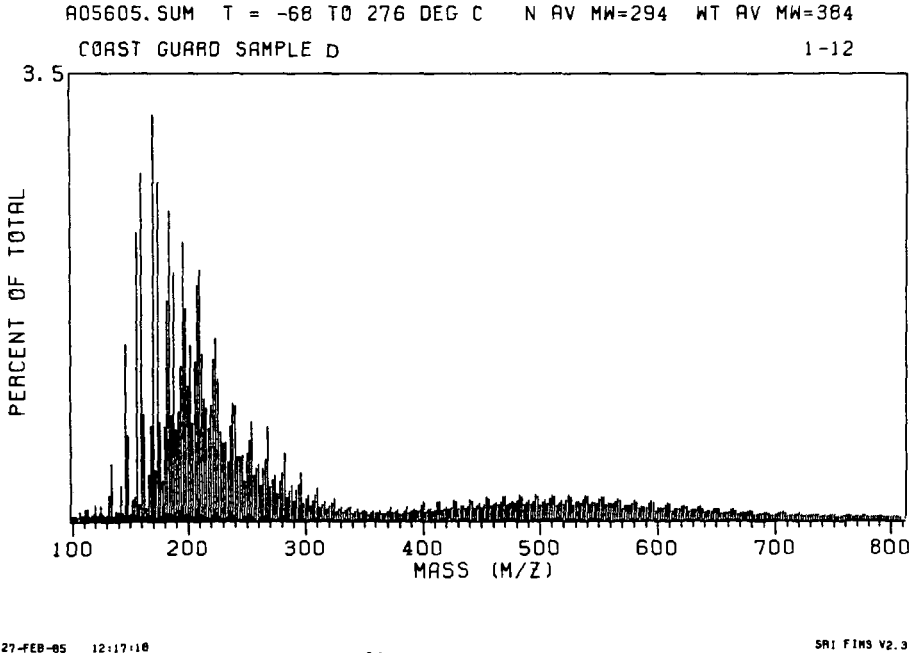
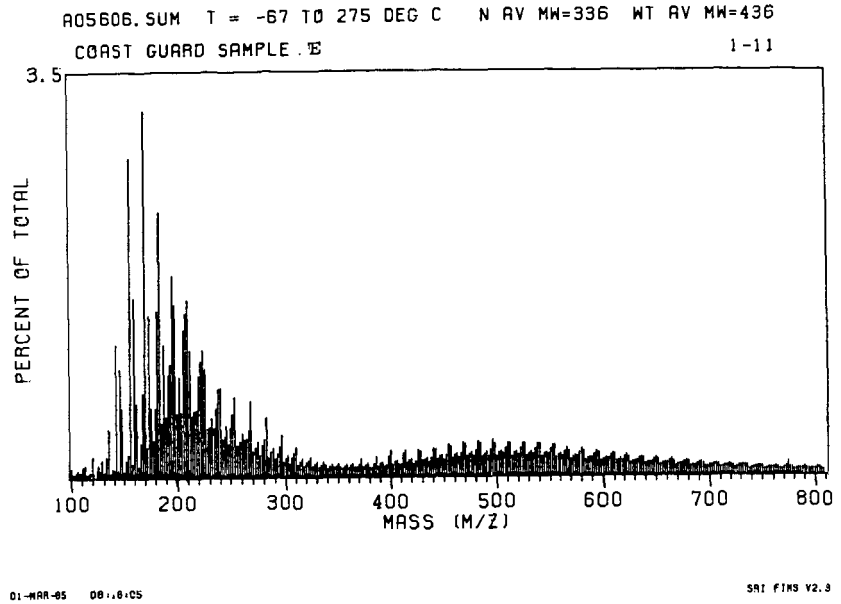
SRI FIMS V2.3

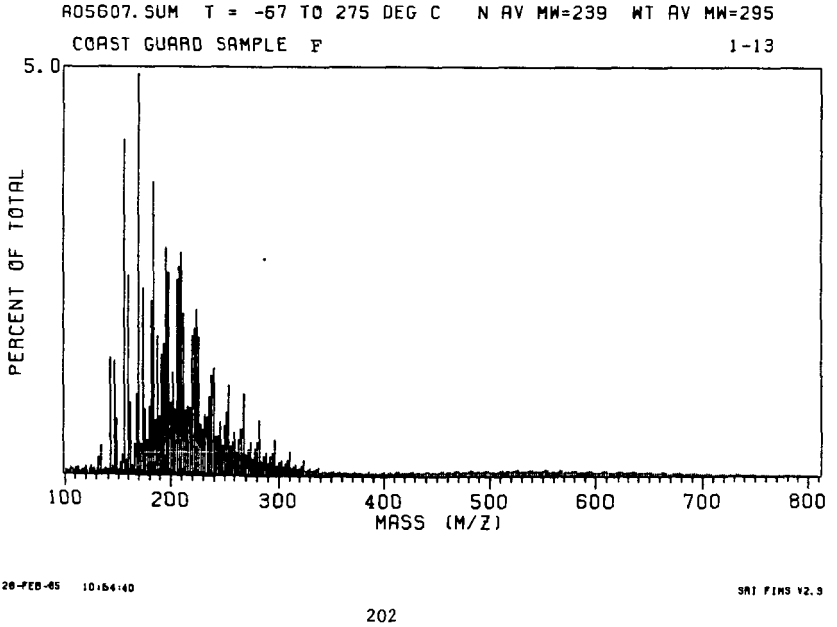

A05601.SUM T = -66 TO 275 DEG C N AV MW=345 WT AV MW=443
COAST GUARD SAMPLE A
1- 9
3.5
PERCENT OF TOTAL
100
200
300
400
500
600
700
800
MASS (M/Z)
28-FEB-85 15:29:42
SRI FIMS V2.3

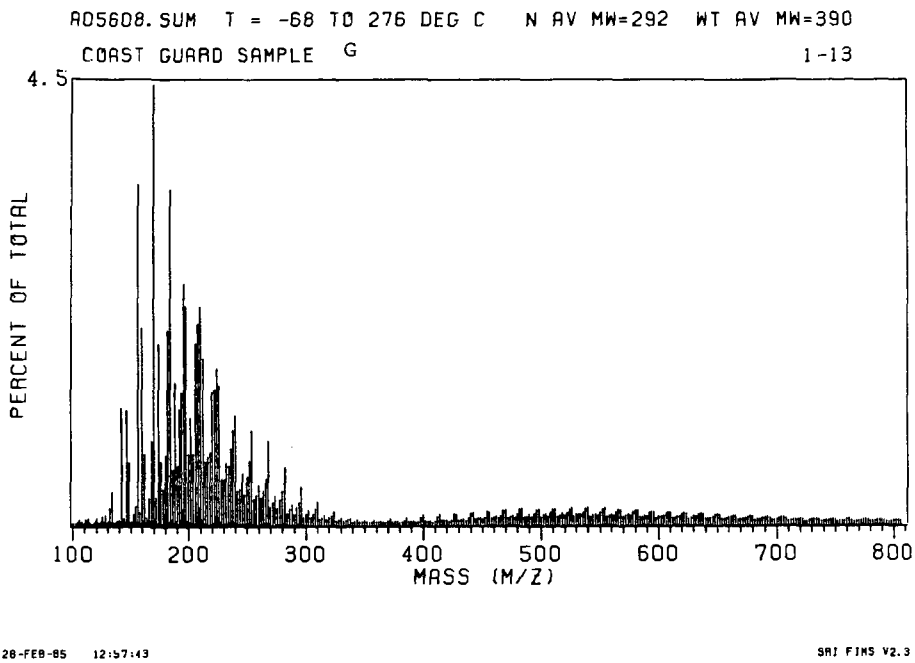

A05603.SUM T = -66 TO 273 DEG C N AV MW=294 WT AV MW=386
COAST GUARD SAMPLE B
1- 9
4.5
PERCENT OF TOTAL
100
200
300
400
500
600
700
800
MASS (M/Z)
01-MAR-85 08:53:49
SRI FIMS V2.3


B05604.SUM T = -64 TO 276 DEG C N AV MW=322 WT AV MW=419
COAST GUARD SAMPLE C
1-11
4.5
PERCENT OF TOTAL
100
200
300
400
500
600
700
800
MASS (M/Z)
27-FEB-85 10:07:27
SRI FIMS V2.3


A05605.SUM T = -68 TO 276 DEG C N AV MW=294 WT AV MW=384
COAST GUARD SAMPLE D
1-12
3.5
PERCENT OF TOTAL
100
200
300
400
500
600
700
800
MASS (M/Z)
27-FEB-85 12:17:18
SRI FIMS V2.3


A05606.SUM T = -67 TO 275 DEG C N AV MW=336 WT AV MW=436
COAST GUARD SAMPLE E
1-11
3.5
PERCENT OF TOTAL
100
200
300
400
500
600
700
800
MASS (M/Z)
01-MAR-85 08:18:05
SRI FIMS V2.3


A05607.SUM T = -67 TO 275 DEG C N AV MW=239 WT AV MW=295
COAST GUARD SAMPLE F
1-13
5.0
PERCENT OF TOTAL
100
200
300
400
500
600
700
800
MASS (M/Z)
28-FEB-85 10:54:40
SRI FIMS V2.3


AD5608.SUM T = -68 TO 276 DEG C N AV MW=292 WT AV MW=390
COAST GUARD SAMPLE G
1-13
4.5
PERCENT OF TOTAL
100
200
300
400
500
600
700
800
MASS (M/Z)
28-FEB-85 12:57:43
SRI FIMS V2.3